INFORMATION SHEET ON THE PREPARATION OF A LIQUID APPLE PECTIN CONCENTRATE

Western Regional Research Laboratory, Albany, California
Bureau of Agricultural and Industrial Chemistry
Agricultural Research Administration
U. S. Department of Agriculture

The purpose of this information sheet is to supply specific information on various phases of the small-scale manufacture of a liquid apple pectin concentrate to members of the preserving and apple-processing industries. Rooker ("Fruit Pectin--Its Commercial Manufacture and Uses," Avi Publishing Co., Inc., New York, 1928) has described the preparation of a similar product and in addition discusses relative sizes, permissible materials of construction, and arrangements of various units for a given scale of manufacture. The method herein described consists of simplified procedures and takes into consideration current practices and requirements.

In work at this Laboratory, special consideration has been given to a method of evaluating samples of dried apple pomace. Tests have been conducted also on the removal of soluble solids, lead, and arsenic from the pomace, the hydrolytic extraction of the pectin to give the maximum yield of jelly units under conditions not too corrosive to metallic equipment, the removal of pulp by pressing, the clarification of the liquor, and finally to concentration and standardization to a definite jellying power.

Evaluation of Dried Apple Pomace

Apple pomace should be evaluated before use, because the pectin content varies from approximately 8 to 11 percent and its quality or jellying power may differ with the variety and stage of maturity of the apple from which the pomace was made. The techniques used in the drying of apple pomace commercially differ, and some processors may have subjected their product to treatments which have materially reduced the jellying power of the pectin contained therein.

Yield of pectin.--To 200 g. of dry apple pomace in 2,000 ml. of water add 5 ml. of 6N sulfuric acid and heat the mixture at just below its boiling point for 1 hour. Filter aid[1] (1 percent by weight) is added and the hot mixture filtered with suction on a canvas precoated with filter aid. The clear filtrate is evaporated to less than 500 ml. under reduced pressure at a temperature below 60° C. The concentrate is diluted to 500 ml. and evaluated for jellying power.

Determination of grade[2].

1. Preparation of standard jelly.--One hundred and sixty-two grams of sugar is needed to give 250 g. of finished standard jelly with a sugar concentration of 65 percent. A sample of pectin of known jellying power equal

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1/ Johns-Manville Hyflo Super-Cel, Dicalite 20, or Dicalite Special Speedflow.

2/ The grade of a pectin has been defined as the weight of sugar with which one part by weight of pectin will form a standard jelly containing 65 percent sugar solids under suitable conditions of acidity.

to ___162___ is weighed to the nearest hundredth of a gram, mixed dry with
known grade
a portion of the sugar equal to approximately 8 times its weight, and the mixture is sprinkled slowly with stirring into 110 ml. of water at 70° C. (158° F.), and then brought to a brisk boil until the pectin is thoroughly dispersed.3/ This temperature is maintained while the remainder of the sugar is slowly added. The jelly mixture is cooked to a weight of 250 g., cooled to 95° C. (203° F.), skimmed, and poured with rapid stirring into an 8-oz. jelly glass containing 2 ml. of standard jelly makers' acid. (Standard jelly makers' acid is prepared by dissolving 227 g. of tartaric or 454 g. of citric acid in 480 ml. of hot water.) The jelly is allowed to cool for 18 hours or more prior to testing.

2. Preparation of experimental jelly.--In the preparation of an experimental jelly for the purpose of evaluating the pectin concentrate obtained from the apple pomace, one can assume it to be 5-grade on the first trial, which means that 162/5 or 32.4 g. of the pectin concentrate is required. The jelly is prepared exactly as described above except that 80 ml. of water is added to the 32.4 g. of pectin sirup, making a total of about 110 ml. of solution. The sugar should be added very slowly while the pectin solution is heated; otherwise the mixture may gel in the jelly pan. The finished jelly is graded by comparing it with the standard jelly either by the finger test method or by other suitable methods which measure to some degree its firmness, resiliency, and texture. If the jelly is much weaker than the standard, a batch should be made wherein the amount of pectin is increased to allow for a lower jelly grade. If the experimental jelly is found to be much firmer than the standard, the amount of pectin should be decreased.

Specific directions, followed by a discussion of the various phases of the process, are presented below:

Preparation of a Liquid Pectin Concentrate

Leaching of pomace.--To one part by weight of dried apple pomace is added 10 parts of water at 50° C. (122° F.) and the mixture slowly stirred for 30 minutes, after which the leach water is drained off and discarded. This treatment should be repeated at least once.

Extraction of the pectin.--To the drained leached pomace is added a quantity of water equivalent to 10 times the weight of the dried pomace. Sufficient sulfuric acid is added to give a pH of about 2.8. The mixture is then heated to boiling and maintained at this temperature for 1 hour by blowing steam through it, which also serves to agitate the mass.

Pressing of the extraction mixture.--To the hot pulpy extraction mixture are added with stirring 0.5 percent by weight each of paper pulp and filter aid.

3/ Pectin of known jellying power is obtainable from commercial manufacturers.

The mass is pressed in a hydraulic press through medium filter cloths[4] or bags at a temperature of 80° C. (176° F.) or above.

Clarification of the extract.--The liquor from the pressing operation is cooled to approximately 50° C. (122° F.) and the pH adjusted to 4.5 with sodium carbonate. Dry enzyme preparations[5] have been found satisfactory when used in concentrations from 0.1 to 0.2 percent by weight of the extract. The temperature is maintained at 50° C. (122° F.) for one to two hours or until a small sample of the liquor no longer gives a blue color when filtered clear and tested with a few drops of 0.1 percent iodine solution. To the pectin extract is added 0.3 percent by weight of citric acid to lower the pH of the solution to about 3.0. The liquor is heated to 80° C. (176° F.) or above, followed by the addition of 0.8 to 1 percent of filter aid and filtered through canvas precoated with filter aid in any suitable filter press. To the hot acidic solution, gaseous sulfur dioxide is added from a cylinder until an excess is detected by its odor. Uncombined sulfur dioxide will be removed during concentration.

Concentration of the clarified liquor.--The clarified liquor is concentrated by evaporation in a vacuum pan until a concentration ratio of approximately 4:1 has been attained. The grade of this concentrate will be about 5, and can be adjusted to that grade either by further concentration or by dilution with water or unconcentrated extract.

Packaging of the standardized concentrate.--The 5-grade pectin concentrate is flash-pasteurized at a temperature near 85° C. (185° F.), followed by packaging in suitable containers.

Discussion

Four-fold concentration of the pectin extract results in a like concentration of such components as undesirable soluble solids, arsenic, and heavy metals such as lead. It is therefore advisable to leach the pomace prior to extraction, thus materially reducing the sugar solids and coloring matter which interfere in the preparation of an attractive light-colored concentrate. At the same time the lead and arsenic contents of the pomace are reduced to such a degree that the concentrate will be within the tolerances allowed by the Food and Drug Administration for those metals on apples and pears. A comparison of the solids and ash contents of a commercial concentrate with concentrates prepared in this Laboratory from a commercial apple pomace is shown in table 1. Leaching of the Laboratory-prepared concentrates was varied from no leaching to 3 leachings.

The separation of the pectin liquor from the pulp by pressing offers no difficulties. The addition of paper pulp and filter aid facilitates the pressing operation and yields a clearer press liquor than is obtainable without their use.

4/ 17.5-oz twill or similar cloth.

5/ Royce Chemical Company, Neozyme, or U.S.P. malt diatase.

TABLE 1.—Effect of number of leachings on composition of 5-grade pectin concentrates[1/]

Sample	Number of leachings	Solids	Ash	Lead[2/]	Arsenic[2,3/]
		percent		ppm.	
Commercial concentrate	—	10.6	0.54	1.5	3.4
WRRL concentrate	0	11.9	0.55	2.0	4.8
" "	1	6.9	0.42	1.2	3.4
" "	2	4.9	0.35	1.2	2.8
" "	3	4.3	0.32	1.2	2.3

1/ Percentages and parts per million on basis of the concentrate.
2/ Tolerances of Food and Drug Administration on apples and pears are: 0.05 grain per pound (7.2 ppm.) for lead and 0.025 grain per pound (3.6 ppm.) for arsenic (arsenic trioxide).
3/ Expressed as arsenic trioxide.

The hydrolytic extraction of pectin from apple pomace with dilute acid is preferable to extraction at the natural acidity of the pomace because of the larger yields and higher quality of the pectin obtained by the former. A comparison of the pectins obtained under the two sets of conditions is shown in Table 2.

TABLE 2.—Effect of pH on yield and quality of pectin[1/]

Extraction media	pH	Time	Temp.	Yield	Jelly grade	Jelly units/100 g. pomace (yield x grade)
		hr.	°C.	%		
Sulfuric acid (dilute)	2.8	1	95	9.5	140	1300
Water	4.0	1	95	5.0	120	600

1/ Pectin isolated by alcohol precipitation evaluated on the dry basis.

Maximum yields of jelly units are obtained by carrying out the extraction at boiling temperature either at a pH of 2.8-3.2 for 1 hour or at lower pH for shorter periods. The advantage of a shorter extraction period is counterbalanced by the disadvantage of increased corrosive action of the media on processing equipment. The effect of the addition of various acids on the pH of a 1:10 mixture of apple pomace and water is shown in Table 3.

TABLE 3.--Effect of additions of acid on the pH of extraction mixtures.

Acid	Ml. acid added to 200 g.[1] pomace dispersed in 2000 ml. water	pH
None	0	4.0
Sulfuric (1:5)	3	3.2
" "	4	3.0
" "	5	2.9
Hydrochloric (conc.)	1	3.2
" "	2	3.0
" "	3	2.8
Citric (standard jelly makers')	5	3.2
" " " "	10	3.0
" " " "	15	2.8

[1] A 200-g. sample of dry pomace was leached twice prior to dispersion.

A number of factors must be considered in the clarification of the press liquor. Starch in the extract will cause turbidity on standing and this condition becomes more pronounced on concentration. The starch can be removed by hydrolysis with diastatic enzymes under certain conditions. A number of commercial powdered enzyme preparations have been found satisfactory for this purpose. A number of them, prepared from molds or bacteria, contain a pectinase which causes degradation of the pectin molecule with subsequent loss in its jellying power. Further clarification of the liquor is accomplished by filtration. Filter presses with wood plate and frames capable of being precoated with a filter aid are suitable for this operation. Pectin liquors are colloidal and viscous but no particular difficulties will be encountered if filter aid is used and the temperature maintained at about 80° C. (176°F.).

Treatment with sulfur dioxide yields a lighter-colored product and is readily accomplished by passing a stream of the gas into the clarified pectin extract until an excess is detected by its odor. There are other ways to add sulfur dioxide which may be more suitable in certain instances and will be equally effective. On concentration of the acidified pectin solution the unconbined sulfur dioxide will be removed.

Concentration *in vacuo* is almost a necessity because of the extensive degradation of the pectin that occurs in open-kettle boiling. For example, a 4:1 concentrate prepared by open-kettle evaporation had a jelly grade of 2.5, while a comparable one prepared by evaporation *in vacuo* had a jelly grade of 5.

A liquid pectin concentrate is most useful if standardized to grade 5, since the viscosity is not too high to allow for ready handling and the volume required for jelly formation is low enough to permit the addition of the necessary amounts of fruit and sugar. It is desirable to overconcentrate slightly and standardize down to grade. Directions for use of the product will be much

simpler if it is standardized, since operators are already familiar with handling commercial preparations of this grade.

The finished concentrate is flash-pasteurized at a temperature near 85°C. (185°F.) and packaged in suitable containers.

A number of apple processors who have quantities of cores and peels available have expressed an interest in the possibility of processing them for pectin without drying. It is not advisable to use fresh material in this process. Fresh apple pulp disintegrates very readily when heated and in so doing swells and absorbs water to such a degree that the pulpy mass cannot be efficiently stirred or pumped unless there is added approximately 3 times the amount of water required for an equivalent weight of the dried pomace. Furthermore, a leaching of the soluble solids and heavy metals is essential with fresh as well as with dried pomace but the leach water in the case of the latter can be drained off while that from the former must be removed by pressing. This additional pressing operation combined with the increased cost of processing 3 times the volume of pectin liquor is not economical.

Those interested in the manufacture of pectin by this process are invited to communicate with or visit the Western Regional Research Laboratory to discuss details or special problems as they arise.